MISSOURI DEPARTMENT OF NATURAL RESOURCES
AIR AND LAND PROTECTION DIVISION
ENVIRONMENTAL SERVICES PROGRAM
Standard Operating Procedures

SOP #: MDNR-FSS-001 EFFECTIVE DATE: September 17, 2003

SOP TITLE: Required/Recommended Containers, Volumes, Preservatives, Holding Times, and Special Sampling Considerations

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SUMMARY OF REVISIONS: Tables I and II were updated and merged to form Appendix A. Appendix B – Percent Ionized (Toxic) Ammonia in Aqueous Solutions chart was deleted. Minor grammatical changes. Organic analyses parameters were updated.

APPLICABILITY: The procedures outlined in this SOP apply to all department personnel or other persons collecting water, soil, sediment, or sludge samples to be analyzed by the ESP or a laboratory contracted by the ESP.

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1.0 SCOPE AND APPLICABILITY

Because of the potential cost to human health and the environment due to poor quality data, it is important that the procedures within this Standard Operating Procedure (SOP) be followed to ensure the quality of samples collected by department personnel. The guidance given in the SOP applies to all department personnel or other persons collecting water, soil, sediment, sludge, hazardous waste, or biological samples for analysis by the Environmental Services Program (ESP).

1.1 The quality of the results of a laboratory analysis can be no better than the quality of the sample to be analyzed. Because of this, it is extremely important that samples be collected and handled in a manner that is consistent with accepted procedures.

1.2 One of the many problems facing field personnel when collecting a sample is to collect and transport to the laboratory a sample that is representative of the material to be analyzed.

1.3 MDNR-FSS-005 *General Sampling Considerations Including the Collection of Grab, Composite, and Modified Composite Samples from Streams and Wastewater Flows*, will aid in proper sample collection of water and wastewater samples. ESP's list of SOPs contains many sampling procedures for specific parameters or matrices that will aid in proper sample collection.

2.0 SUMMARY OF METHOD

The procedures described within this SOP provide guidance for the sample collector by providing tables to assist the sampler to more easily determine the volume of sample needed, the correct containers and preservatives, and the allowable holding times. The guidance within this SOP is consistent with, and conforms to, the following references: Standard Methods for the Examination of Waters and Wastewater, 20th edition; 40 Code of Federal Regulations, 136.3; 10 Code of State Regulations, Division 20, Chapter 7; and Methods for Chemical Analysis of Water and Wastes (EPA-600/4-79-020), Test Methods for Evaluations of Solid Waste, Physical/Chemical Methods (SW-846). The procedure also discusses safety issues and personnel qualifications that need to be addressed when collecting samples.

3.0 HEALTH AND SAFETY

3.1 Chemical preservatives frequently consist of concentrated acid or base. The sample collector must have a basic understanding of the hazards associated with the handling of these chemicals. Personnel should read applicable Material Safety Data Sheets for specific health and safety information on chemical preservatives that are used.

3.2 The sample collector shall use an appropriate level of personal protection based on the specific work being done. The minimum level of personal protection to be used is protective gloves and safety glasses. A more stringent level of protection may be required, such as those outlined in a site specific Health and Safety Plan, etc.

4.0 PERSONNEL QUALIFICATIONS

Personnel who handle samples and preservatives shall:

- Receive appropriate on-the-job training.
- Be familiar with the ESP SOP manual and have read all SOP documents that are applicable to their assigned activities.
- Participate in the departmental medical monitoring program in accordance with department policy.

5.0 PROCEDURES

5.1 Handling and Preservation Recommendations for Water, Solids, Sediment, and Sludge (Appendix A).

In order to ensure that the sample reaching the analytical laboratory is a representative one, the collector should consider:

- Sample containers - The types of sample containers approved by the ESP are ones that meet guidelines established by EPA or Standard Methods or have been shown through ESP tests to be appropriate. Because not all glass is alike and not all plastic is alike, only those containers meeting ESP approval will be accepted by the ESP laboratory. In general ESP approved containers will be precleaned, precleaned/certified or otherwise tested to insure cleanliness. Containers from unproven sources or of unknown cleanliness will be rejected by the ESP laboratory.

- Preservatives - Ideally, samples should be analyzed immediately upon collection. Since this is not usually possible, steps need to be taken to retard chemical and biological activity that could alter a sample while in storage. Methods of preservation are generally limited to pH adjustment, chemical addition, and cooling using ice or refrigeration. Samples requiring preservation should be preserved immediately upon collection.

- Sample Labels - All sample containers must be labeled with department sample labels indicating the preservative used, the date and time of sample collection, and the collector's initials. Information requested on

the label should be recorded according to the guidelines provided in MDNR-FSS-003 *Sample Numbering and Labeling*.

- Sample Volumes - Adequate sample volume/weight for a specific analysis is important. The optimum volume/weight is listed for each parameter in Appendix A. When possible this amount should be collected, so, if needed, several "runs" can be done for a particular parameter. The additional sample amount also allows for quality control checks, etc., improving data reliability. Less than the optimum volume/weight can be submitted. If however, a portion of that sample is lost due to spillage, machine malfunction, operator error, etc., and there is not enough remaining to perform the analysis, the laboratory will necessarily report a No Result for that parameter.

- Holding Time - Holding time is the maximum amount of time that samples may be stored before analysis is initiated and still is considered valid. The sample collector must **notify the ESP, CAS prior to sampling** and make sure that samples reach the laboratory as soon as possible after collection so that sample analysis can be initiated before the holding time is exceeded. **Prior notification is particularly vital, when a sample either has a short holding time or when it must be shipped to a contract laboratory for analysis.**

- Special Handling Considerations - Many parameters require special handling or sampling techniques. These are described in the "Notes" located at the end of Appendix A. When analytical techniques are available for field determinations, the corresponding SOPs that describe those techniques are listed in the Notes.

5.2 Analyzing Several Parameters from One Sample Container

5.2.1 It is permissible and often desirable to have more than one parameter analyzed from the same sample container. This is generally acceptable if the following conditions are met:

- Preservatives, containers, and other handling requirements are the same.
- The sum of the optimum volume/weight for each parameter does not exceed the capacity of the sample container.
- All analyses requested from one sample container are to be analyzed at the same laboratory.

5.2.2 Examples of parameters that are commonly combined into one container:

- TDS, fluoride, chloride (1 Cubitainer)

- chloride, sulfate (1 Cubitainer)
- NH_3-N, NO_2 + NO_3, Total P (1 Cubitainer + H_2SO_4)
- COD, NH_3-N (1 Cubitainer + H_2SO_4)

NOTE: Samples for organic constituents should not be combined for analyses.

5.3 Trip Blanks and Other Quality Control Procedures

5.3.1 Trip blanks are ESP-prepared containers filled with ESP analyte-free water. For soil samples, a commercially prepared soil trip blank may be purchased. Trip blanks consist of containers that are of the same type and handled in the same manner as those that will be used for sample collection. Trip blanks accompany the sample containers and samples but remain closed during the entire sampling trip. Any required preservatives are added when the trip blank is prepared (refer to MDNR-FSS-210 *Quality Assurance/Quality Control for Environmental Data Collection).*

5.3.2 To determine if a trip blank is required, consult Appendix A under the column "Notes." In general, trip blanks are required for all organic analyses of water samples submitted to the ESP.

5.3.3 When required, each <u>set</u> of samples collected should be accompanied by an appropriate blank.

For example:

- If one sample is collected for pesticide analysis, it should be accompanied by a trip blank.

- If, in one day, six samples are collected for pesticide analyses, the set of six should be accompanied by one trip blank. If the samples are collected in two successive days, each days samples should be accompanied by a separate trip blank.

- If one sample is collected for pesticide analysis and one sample is collected for volatile organic analysis (VOA), each should be accompanied by an appropriate trip blank.

5.3.4 Trip blanks or other quality control procedures for organic or other parameters may be requested at the discretion of the ESP, the clients, or required due to specific regulatory requirements. Please refer to MDNR-FSS-210 *Quality Assurance/Quality Control for Environmental Data Collection* for further discussion of proper quality control procedures.

6.0 REFERENCES

MDNR-FSS-005 *General Sampling Considerations Including the Collection of Grab, Composite, and Modified Composite Samples from Streams and Wastewater Flows*

Standard Methods for the Examination of Water and Wastewater, 1998, 20th Edition

40 Code of Federal Regulations, 136.3

10 Code of State Regulations, Division 20, Chapter 7

Methods for Chemical Analysis of Water and Wastes, EPA-600/4-79-020

Test Methods for Evaluations of Solid Waste, Physical/Chemical Methods, SW-846

MDNR-FSS-003 *Sample Numbering and Labeling*

MDNR-FSS-210 *Quality Assurance/Quality Control for Environmental Data Collection*

MDNR-FSS-102 *Field Analysis of Specific Conductance*

MDNR-FSS-101 *Field Measurement of Water Temperature*

MDNR-FSS-100 *Field Measurement of pH*

MDNR-FSS-103 *Sample Collection and Field Analysis for Dissolved Oxygen Using a Membrane Electrode Meter*

MDNR-FSS-108 *Field Analysis of Fecal Coliform*

MDNR-FSS-015 *Sample Collection and Field Handling Procedures for Chlorophyll Analysis of Surface Water Samples*

MDNR-FSS-006A Sampling Water and Other Liquids for Volatile Organic Analysis (VOA).

APPENDIX A

Environmental Services Program for Sampling Preservation of Water, Solids, Sediment and Sludge

Environmental Services Program for Sampling Preservation of Water, Solids, Sediment and Sludge

Parameter	Sample Matrix *	Optimum Volume	Container Type**	Preservation Method	Holding Time ***	Notes
PHYSICAL PROPERTIES						
Filterable Residue also called Total Dissolved Solids (TDS)	**W**	500 mL	P,G	Cool	7 days	
Non Filterable Residue (NFR) also called Total Suspended Solids (TSS)	**W**	500 mL	P,G	Cool	7 days	
Settleable Solids (SS)	**W**	500 mL	P,G	Cool	48 hours	
Specific Conductivity	**W**	1000 mL	P,G	Cool	28 days	1
Temperature	**W**	Determine on-site			None	2
Total Solids	**W**	500 mL	P,G	Cool	7 days	
Turbidity (NTU)	**W**	500 mL	P,G	Cool	48 hours	
Volatile Suspended Solids (VSS)	**W**	500 mL	P,G	Cool	7 days	
INORGANIC CONSTITUENTS						
Acidity (Acid.)	**W**	500 mL	P,G	Cool	14 days	
Alkalinity (ALK.)	**W**	500 mL	P,G	Cool	14 days	3
Bromide (Br)	**W**	500 mL	P,G	Cool	28 days	
Chloride (Cl)	**W**	500 mL	P,G	Cool	28 days	
Cyanide (CN), Total	**W**	1000 mL	P,G	Cool, NaOH to pH > 12	28 days	4
	S	8 oz jar	G	Cool	28 days	
Cyanide (CN), Amenable to Chlorination	**W**	1000 mL	P,G	Cool, NaOH to pH > 12	28 days	4
Fluoride (F)	**W**	500 mL	P,G	Cool	28 days	
Hardness	**W**	500 mL	P,G	Cool, HNO_3 to pH < 2	6 mos.	5

*Sample Matrix Labels: **A** = Air, **W** = Water (As found in 40 CFR 136.3), **S** = Solids, Sediments, and Sludge
Container types: **P = Plastic, **G** = Glass, or as specified within the table
***The Holding Time is the maximum time a sample can be held before analysis is started

Environmental Services Program for Sampling Preservation of Water, Solids, Sediment and Sludge

Parameter	Sample Matrix *	Optimum Volume	Container Type**	Preservation Method	Holding Time ***	Notes
Hydrogen ion (pH)	**W**	100 mL	P,G	Determine on-site	None	6,7
Nitrogen Ammonia, Total (NH_3 as N)	**W**	300 mL	P,G	Cool, H_2SO_4 to pH < 2	28 days	8
Nitrogen Ammonia, Unionized (unionized NH_3 as N)	**W**	Calculated using NH_3 as N, pH & Temp.				9
Nitrogen, Total (TN)	**W**	Calculation: TKN plus (NO_2^- + NO_3^- as N)				
Nitrogen, Total Kjeldahl (TKN)	**W**	1000 mL	P,G	Cool, H_2SO_4 to pH < 2	28 days	10
	S	8 oz. jar	G	Cool	28 days	10,18
Nitrogen as Nitrate (NO_3^- as N)	**W**	Calculation: (NO_2^- + NO_3^- as N) minus (NO_2^- as N)				11
Nitrogen as Nitrite (NO_2^- as N)	**W**	500 mL	P,G	Cool	48 hrs.	
Nitrogen as Nitrite + Nitrate (NO_2^- + NO_3^- as N)	**W**	300 mL	P,G	Cool, H_2SO_4 to pH < 2	28 days	
	S	8 oz jar	G	Cool	28 days	
Nitrogen as Total Organic Nitrogen as N (TON)	**W**	Calculation: TKN minus T(NH_3)				12
Oxygen, Dissolved – Probe	**W**	Determine on-site			None	13
Oxygen, Dissolved – Winkler	**W**	300 mL	G	Fix on-site	8 hrs.	14
Phosphorus, Dissolved Orthophosphate	**W**	250 mL	P,G	Filter on-site; Cool	48 hrs.	15
Phosphorus, Total as P	**W**	250 mL	P,G	Cool, H_2SO_4 to pH < 2	28 days	
	S	8 oz jar	G	Cool	28 days	

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Environmental Services Program for Sampling Preservation of Water, Solids, Sediment and Sludge

Parameter	Sample Matrix *	Optimum Volume	Container Type**	Preservation Method	Holding Time ***	Notes
Metals, Total (Ag, As, B, Ba, Be, Ca, Cd, Co, Cr, Cu, Fe, K, Li, Mn, Mo, Na, Pb, Ni, Zn, Al, Sb, Sr, Ti, Tl, Se, Sn, Mg, Hg, V)	**W**	1000 mL	P,G	Cool, HNO_3 to pH < 2	6 mos. (28 days for Hg)	
	S	8 oz. jar	G	Cool	6 mos.	
Metals, Total Recoverable	**W**	1000 mL	P,G	Cool, HNO_3 to pH < 2	6 mos. (28 days for Hg)	
Metals, Dissolved (Ag, As, B, Ba, Be, Ca, Cd, Co, Cr, Cu, Fe, K, Li, Mn, Mo, Na, Pb, Ni, Zn, Al, Sb, Sr, Ti, Tl, Se, Sn, Mg, Hg, V)	**W**	1000 mL	P,G	Filter on-site; Cool, HNO_3 to pH < 2	6 mos. (28 days for Hg)	15
Metals, Dissolved Hexavalent Cr	**W**	1000 mL	P,G	Filter on-site; Cool	24 hrs.	15
Metals, TCLP *(refer to Appendix C)*	**W**	2000 mL	P,G	Cool	6 mos. (28 days for Hg)	
	S	(2) 8 oz jars	G	Cool	6 mos. (28 days for Hg)	
Sulfate (SO4)	**W**	250 mL	P,G	Cool	28 days	
ORGANIC CONSTITUENTS (Note 16)						
Atrazine Compounds Method 507/508	**W**	2000 mL	G	Cool	7 days to extract	18,20
Ethylene Glycol (antifreeze)	**W**	(2) 40 mL VOA vials	G	Cool	14 days	18, 19, 20
Chemical Oxygen Demand (COD)	**W**	500 mL	P,G	Cool, H_2SO_4 to pH < 2	28 days	
Flashpoint	**W**	2 oz jar	G	Cool	Indefinite	
Nitroglycerine (NG) and Ethylene Glycol Dinitrate (EGDN)	**W**	2000 mL	G	Cool	7 days to extract	18,20
Oil and Grease	**W**	2000 mL	G	Cool, H_2SO_4 to pH < 2	28 days	6,21

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Environmental Services Program for Sampling Preservation of Water, Solids, Sediment and Sludge

Parameter	Sample Matrix *	Optimum Volume	Container Type**	Preservation Method	Holding Time ***	Notes
Total Organic Carbon (TOC)	**W**	(2) 40 mL VOA vials	G	Cool, H_3PO_4 to pH < 2	28 days	6,17,18
	S	8 oz jar	G	Cool	28 days	18
Total Organic Halides (TOX)	**W**	2000 mL	G	Cool, H_2SO_4 to pH < 2	7 days to extract	18,19,20
Total Petroleum Hydrocarbons (TPH)	**W**	2000 mL	G	Cool, HCl or H_2SO_4 to pH < 2	28 days	6,18,20
	S	8 oz jar	G	Cool	28 days	18
Total Phenols	**W**	2000 mL	G	Cool, H_2SO_4 to pH < 2	28 days	20
Polynuclear Aromatic Hydrocarbons (PAH)	**W**	2000 mL	G	Cool	7 days to extract	6,18,20
	S	8 oz jar	G	Cool	14 days to extract	18, 22
Total Toxic Organics (TTO) Analysis includes: acid extractables, base neutrals, PCB's, Pesticides, and VOA						22
Base Neutrals/Acid Extractables (BNA) Analysis includes: Base neutrals and Acid extractables						22
Acid extractables *(refer to Appendix B)*	**W**	2000 mL	G	Cool	7 days to extract	18,20,22
	S	8 oz jar	G	Cool	14 days to extract	18
Acid extractables, TCLP *(refer to Appendix C)*	**S**	(2) 8 oz jars	G	Cool	28 days	
Base neutrals Method 8270C	**W**	2000 mL	G	Cool	7 days to extract	18,20,22
	S	(2) 8 oz jars	G	Cool	14 days to extract	18
Base neutrals, TCLP *(refer to Appendix C)*	**S**	(2) 8 oz. jars	G	Cool	28 days	
Chlorinated Herbicides (*refer to Appendix C)*	**S**	8 oz jar	G	Cool	28 days	

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Container types: **P = Plastic, **G** = Glass, or as specified within the table
***The Holding Time is the maximum time a sample can be held before analysis is started

Environmental Services Program for Sampling Preservation of Water, Solids, Sediment and Sludge

Parameter	Sample Matrix *	Optimum Volume	Container Type**	Preservation Method	Holding Time ***	Notes
Chlorinated Herbicides, TCLP *(refer to Appendix C)*	**S**	8 oz jar	G	Cool	28 days	
PCB's *(refer to Appendix B)*	**W**	2000 mL	G	Cool	7 days to extract	18,20
	S	(2) 8 oz jars	G	Cool	14 days to extract	18
Organochlorine Pesticides *(refer to Appendix B) Method 8081A* **or** *Organophosphorus Compounds (Chlorpyrifos and Diazinon) Method 8141A*	**W**	2000 mL	G	Cool	7 days to extract	18,20
	S	(2) 8 oz jars	G	Cool	14 days to extract	18
Organochlorine Pesticides, TCLP *(refer to Appendix C) Method 8081A*	**S**	8 oz jar	G	Cool	28 days	
Volatile organic analyses (VOA) Method 8260 *(refer to Appendix B)*	**W**	(2) 40 mL VOA vials	G	Cool, HCl to pH < 2	14 days	6,17,18,20, 30,33
	S	(4) 5-gram EnCore	EnCore Sample	Cool	48 hours	31
	S	(2) 2 oz jars	G	Cool	14 days	
Volatile organic analyses, TCLP *(refer to Appendix C)*	**W**	(2) 40 mL VOA vials	G	Cool	14 days	6,18,20,30, 33
	S	(2) 5 gram EnCore	EnCore Sample	Cool	48 hours	31
	S	25 gram EnCore	EnCore Sample	Cool	48 hours	31
	S	2 oz. jar	G	Cool	14 days	
Volatile organic compound (VOC) Method 524.2	**W**	(2) 40 mL VOA vials	G	Cool, HCl to pH < 2	14 days	6,17,18,20, 30
OA1 (BTEX) Method 8260B	**W**	(2) 40 mL VOA vials	G	Cool, HCl to pH < 2	14 days	6,17,18,20
	S	(4) 5-gram EnCore	EnCore Sample	Cool	48 hours	31
	S	(2) 2 oz jars	G	Cool	14 days	32

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Container types: **P = Plastic, **G** = Glass, or as specified within the table
***The Holding Time is the maximum time a sample can be held before analysis is started

Environmental Services Program for Sampling Preservation of Water, Solids, Sediment and Sludge

Parameter	Sample Matrix *	Optimum Volume	Container Type**	Preservation Method	Holding Time ***	Notes
OA2 (Petroleum Fractions) Method 8015	**W**	3000 mL	G	Cool	7 days to extract	6,18,20
	S	8 oz jar	G	Cool	14 days	32
2,3,7,8 - Tetrachloro-dibenzo-P-Dioxins (TCDD)	**W**	2000 mL	G	Cool	30 days to extract	18,20
	S	8 oz jar	G	Cool	30 days to extract	18
BIOLOGICAL and AIR PARTICULATES						
Algal I.D.	**W**	500 mL	P,G	Cool or 10% formalin or Lugols	48 hrs. or Indefinite	26
Air Particulate I.D.	**A**	10 grams	P,G	None	Indefinite	28
Asbestos	**A**	10 grams	P,G	None	Indefinite	28,29
BOD (grab)	**W**	1000 mL	P,G	Cool	48 hrs.	
BOD (composite)	**W**	1000 mL	P,G	Cool	36 hrs.	23
CBOD (grab)	**W**	1000 mL	P,G	Cool	48 hrs.	
CBOD (composite)	**W**	1000 mL	P,G	Cool	36 hrs.	23
Chlorophyll a	**W**	20-100 mL	P,G	Filter on-site; freeze in desiccant	1 month	25
Fecal Coliform or E. coli	**W**	150 mL	Sterile bacteriological bottle	Cool	6 hrs.	6,24,30
Microtox toxicity	**W**	100 mL	P,G	Cool	48 hrs	27

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Container types: **P = Plastic, **G** = Glass, or as specified within the table
***The Holding Time is the maximum time a sample can be held before analysis is started

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NOTES

Note 1. Field procedure; see MDNR-FSS-102 *Field Analysis of Specific Conductance* (Record Units on Chain of Custody).

Note 2. Field procedure; see MDNR-FSS-101 *Field Measurement of Water Temperature* (Record Units on Chain of Custody).

Note 3. For field analysis, commercially obtained kits may be used. Follow manufacturer's directions.

Note 4. When total CN and CN amenable to chlorination are both to be analyzed, they must be collected in separate containers.

Note 5. For field analysis, commercially obtained kits may be used. Follow manufacturer's directions. **If requesting hardness to be calculated by the CAS, total, total recoverable or dissolved Ca and Mg must also be requested in addition to hardness on Chain-of-Custody.**

Note 6. Grab sample only.

Note 7. Field procedure; see MDNR-FSS-100 *Field Measurement of pH.*

Note 8. Analytically NH_3^- and NH_4^+ are determined together and expressed as total ammonia as nitrogen. **In highly contaminated samples (e.g. raw livestock manure) that generate gasses when preserved, submittal of an unpreserved sample is permissible.** If such a sample is collected, the CAS should be notified immediately so they can prepare for immediate analysis and the sample should be submitted as soon as possible.

Note 9. This is usually considered the most toxic form of ammonia.

Note 10. Analytically organic nitrogen and ammonia are determined together and expressed as Total Kjeldahl Nitrogen (TKN = TON plus NH_3-N).

Note 11. When nitrate (NO_3^- as N) is requested separately, NO_2^- and NO_2^- + NO_3^- as N must both be requested and the appropriate sample containers must be provided by the sample collector.

Note 12. Total organic nitrogen (TON) is a calculated result that is determined by subtracting the NH_3-N result from the TKN result. Therefore, when organic nitrogen is requested, TKN and NH_3-N must also be requested and sufficient sample provided for analysis (TON = TKN minus NH_3-N). Organic nitrogen is organically bound nitrogen in the tri-negative oxidation state. It includes natural materials as proteins and peptides, nucleic acids and urea, and it is formed from numerous synthetic organic materials.

Note 13. Field procedure; see MDNR-WQMS-103 *Sample Collection and Field Analysis for Dissolved Oxygen Using a Membrane Electrode Meter.*

Note 14. For field preservation and analysis, see MDNR-FSS-103 *Sample Collection and Field Analysis for Dissolved Oxygen Using a Membrane Electrode Meter.*

Note 15. Filter on-site through a 0.45um filter (see Standard Methods, 20th edition) prior to adding any preservative. Write "filtered" on the sample label in the "other" area.

Note 16. The following volume requirements are for samples that are not pure organic compounds. If material is pure, (e.g., oil from transformer, oil spill, solvent from can or drum), consult with Chemical Analysis Section for quantity of sample needed for analysis in order to minimize disposal costs.

Note 17. Add four (4) drops of concentrated HCl acid to each of two (2) 40 mL VOA vials, fill fully with sample and cap in such a manner as to exclude all air bubbles.

Note 18. When collecting a sample for this parameter, additional analyses cannot be performed from the same sample container. Therefore, a separate sample container is needed.

Note 19. Containers must be filled fully and capped in such a manner as to exclude all air bubbles.

Note 20. Trip blank should be collected. See MDNR-FSS-210 *Quality Assurance/Quality Control for Environmental Data Collection,* for more information.

Note 21. As part of an overall effort to reduce the amount of chlorofluorocarbons (CFCs) released into the environment, the use of the standard Freon-113 Method of extraction has been discontinued. It has been replaced with EPA Method 1664 that uses hexane as the extraction solvent. Because oil and grease is a method-defined analyte, the amount of oil and grease in a sample depends on the solvent used for extraction. Sample results obtained using hexane as the extraction solvent may or may not be comparable to those obtained using the freon extraction. Any comparisons between results obtained from different methods of analysis, therefore, should be made with caution.

Note 22. See Appendix B for a list of chemicals included in TTO. PAH's are a subset of the Base Neutral/Acid Extractable analysis. They may be requested separately but may have to be contracted out.

Note 23. Holding time measured from time last aliquot is collected.

Note 24. For field analyses, see MDNR-FSS-108 *Field Analysis of Fecal Coliform.*

Note 25. For field collection and handling, see MDNR-FSS-015 *Sample Collection and Field Handling Procedures for Chlorophyll Analysis of Surface Water Samples.*

Note 26. If sample can be received by laboratory within 48 hours of time of collection, an unpreserved sample is preferred. If not, holding time can be extended by preserving with formalin or Lugol's.

Note 27. Microtox is a rapid, inexpensive bioassay technique, which utilizes fluorescent bacteria to determine the relative toxicity of water, sediment, and soil samples. The results allow for the prediction of how constituents of a sample might affect other living organisms. If the sample has been disinfected with chlorine, it must be indicated on the sample label.

Note 28. Double bag, such as a film canister within a Ziploc bag with sample tag taped to canister. If asbestos is a requested analysis sample, it must be clearly identified as such and separate paperwork (chain-of-custody, etc.) should accompany the sample.

Note 29. All samples to be analyzed for asbestos are currently contracted to a private lab(s) directly through each regional office. ESP does not conduct nor review the results of these analyses.

Note 30. If the sample has been disinfected (chlorine, ultraviolet light, etc.), it shall be indicated on the sample label and noted on the Chain-of-Custody record. If the sample has been chlorinated, sodium thiosulfate must be added to the sample container that is to be submitted for fecal coliform, VOA or VOC analysis. The addition of sodium thiosulfate to a sample container will neutralize any residual chlorine and prevent biological degradation and/or inhibit the formation of chlorinated hydrocarbons.

Note 31. If samples can be frozen within the 48-hour holding time, then the holding time is extended to 14 days.

Note 32. Sample containers should be packed full with no headspace.

Note 33. Field procedure, see MDNR-FSS-006A *Sampling Water and Other Liquids for Volatile Organic Analysis (VOA).*

APPENDIX B

TOTAL TOXIC ORGANICS

APPENDIX B
TOTAL TOXIC ORGANICS

BASE NEUTRALS

N-NITROSODIMETHYL AMINE
ANILINE
BIS(2-CHLOROETHYL) ETHER
1,3-DICHLOROBENZENE
1,4-DICHLOROBENZENE
BENZYL ALCOHOL
1,2-DICHLOROBENZENE
BIS(2-CHLOROISOPROPYL) ETHER
N-NITROSO-DI-PROPYLAMINE
HEXACHLOROETHANE
ISOPHORONE
BIS(2-CHLOROETHOXY) METHANE
1,2,4-TRICHLOROBENZENE
NAPHTHALENE*
4-CHLOROANALINE
HEXACHLOROBUTADIENE
2-METHYLNAPHTHALENE
HEXACHLOROCYCLOPENTADIENE
2-CHLORONAPHTHALENE
2-NITROANILINE
DIMETHYL PHTHALATE
ACENAPHTHYLENE*
3-NITROANILINE
ACENAPHTHENE*
DIBENZOFURAN
2,4-DINITROTOLUENE
2,6-DINITROTOLUENE
DIETHYLPHTHALATE
4-CHLOROPHENYL-PHENYLETHER
FLUORENE*
4-NITROANILINE
N-NITROSODIPHENYLAMINE
4-BROMOPHENYL-PHENYLETHER
HEXACHLOROBENZENE
PHENANTHRENE*
ANTHRACENE*
DI-N-BUTYLPHTHALATE
FLUORANTHENE*
PYRENE*
BUTYLBENZYLPHTHALATE
3,3'-DICHLOROBENZIDINE
BENZO(A)ANTHRACENE*
BIS(2-ETHYLHEXYL)PHTHALATE
CHRYSENE*
DI-N-OCTYL-PHTHALATE
BENZO(B)FLUORANTHENE*
BENZO(K)FLUORANTHENE*
BENZO(A)PYRENE*
INDENO(1,2,3-CD)PYRENE*
DIBENZ(A,H)ANTHRACENE*

VOLATILE ORGANICS

CHLOROMETHANE
BROMOMETHANE
VINYL CHLORIDE
CHLOROETHANE
METHYLENE CHLORIDE
ACETONE
CARBON DISULFIDE
1,1-DICHLOROETHENE
1,1-DICHLOROETHANE
1,2-DICHLOROETHENE
CHLOROFORM
1,2-DICHLOROTHANE
2-BUTANONE
1,1,1-TRICHLOROETHANE
CARBON TETRACHLORIDE
VINYL ACETATE
BROMODICHLOROMETHANE
1,2-DICHLOROPROPANE
TRANS-1,3-DICHLOROPROPENE
TRICHLOROETHENE
DIBROMOCHLOROMETHANE
1,1,2-TRICHLOROETHANE
BENZENE
CIS-1,3-DICHLOROPROPENE
BROMOFORM
2-HEXANONE
4-METHYL-2-PENTANONE
TETRACHLOROETHENE
1,1,2,2-TETRACHLOROETHANE
TOLUENE
CHLOROBENZENE
ETHYLBENZENE
STYRENE
M-XYLENE AND P-XYLENE
O-XYLENE

*Polycyclic Aromatic Hydrocarbons

ACIDS

PHENOL
2-CHLOROPHENOL
2-METHYLPHENOL
4-METHYLPHENOL
2-NITROPHENOL
2,4-DIMETHYLPHENOL
BENZOIC ACIDS
2,4-DICHLOROPHENOL
4-CHLORO-3-METHYLPHENOL
2,4,6-TRICHLOROPHENOL
2,4,5-TRICHLOROPHENOL
2,4-DINITROPHENOL
4-NITROPHENOL
4,6-DINITRO-2-METHYLPHENOL
PENTACHLOROPHENOL

PESTICIDES/PCB's

PCB-1016
PCB-1221
PCB-1232
PCB-1242
PCB-1248
PCB-1254
PCB-1260
ALPHA-BHC
BETA-BHC
GAMMA-BHC
DELTA-BHC
HEPTACHLOR
ALDRIN
HEPTACHLOR EPOXIDE
ALPHA-ENDOSULFAN
DDE
DIELDRIN
ENDRIN
BETA-ENDOSULFAN
DDD
ENDRIN ALDEHYDE
ENDOSULFAN SULFATE
DDT
CHLORDANE
TOXAPHENE

APPENDIX C

TOXICITY CHARACTERISTIC LEACHING PROCEDURE (TCLP)

APPENDIX C
Toxicity Characteristic Leaching Procedure (TCLP)

The following is a list of all the chemical parameters found on the TCLP list, referencing 40 CFR part 261.24:

Metals (8)	Volatile Organics (10)	Pesticides (7)
Arsenic	Benzene	Chlordane
Barium	Carbon Tetrachloride	Endrin
Cadmium	Chlorobenzene	Heptachlor
Chromium	Chloroform	Heptachlor Epoxide
Lead	1,2-Dichloroethane	Lindane
Mercury	1,1-Dichloroethylene	Methoxychlor
Selenium	Methyl Ethyl Ketone	Toxaphene
Silver	Tetrachloroethylene	
	Trichloroethylene	
	Vinyl Chloride	

Base Neutral Organics (7)	Acid Extractable Organics (7)	Herbicides (2)
1,4-Dichlorobenzene	Pentachlorophenol	2,4-D
2,4-Dinitrotoluene	2,4,5-Trichlorophenol	2,4,5-TP (Silvex)
Hexachlorobenzene	2,4,6-Trichlorophenol	
Hexachloro-1,3-butadiene	o-cresol	
Hexachloroethane	m-cresol	
Nitrobenzene	p-cresol	
Pyridine	Cresol	

When a request is made for TCLP analysis, generally the sample will first be analyzed for total concentrations to determine whether the level of contamination in the sample warrants the additional step of performing TCLP analysis. If TCLP analysis is warranted, additional sample volume will be needed. If TCLP analysis is requested, the sample collector will need to request the specific group of parameters desired (from the list above) and will need to collect the proper sample volumes listed in Appendix A for both total and TCLP.